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Acta Cryst. (1994). C**50**, 861-862

(*O*-Ethyl dithiocarbonato-κ*S*)phenylmercury, [Hg($C_3H_5OS_2$)(C_6H_5)]

EDWARD R. T. TIEKINK

Department of Chemistry, The University of Adelaide, Adelaide, SA 5005, Australia

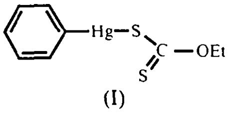
(*Received* 17 *November* 1993; *accepted* 5 *January* 1994)

Abstract

The Hg atom in [PhHg(S_2COEt)] adopts a linear coordination geometry defined by the phenyl C atom [2.06 (1) Å] and an S atom [2.342 (3) Å] such that the C—Hg—S angle is 176.1 (4)°. A weak intramolecular Hg···S contact of 3.209 (4) Å is noted.

Comment

The xanthate ligand ($ROCS_2^-$) is known to adopt a variety of coordination modes (Tiekink & Winter, 1992). For monodentate coordination *via* one of the S atoms, a structural variation may occur such that either the pendent S atom or the O atom may be directed towards the metal centre (Tiekink & Winter, 1992). The different conformations have been termed the *S,S* and *S,O* conformations, respectively. Recent studies on a series of triorganophosphinegold(I) xanthates have shown that both conformations may be found; these have been rationalized in terms of packing efficiency (Siasios & Tiekink, 1993*a*,*b*). While a similar variation of structure is possible for the organomercury xanthates, to date only the *S,S* conformation has been found. It was within the context of these studies that the structure of the title compound (I) was investigated.



(I)

The Hg atom in (I) adopts the expected linear coordination geometry, and is cooordinated by a phenyl C atom and the S(1) atom from a monodentate xanthate ligand. The second S atom is 3.209 (4) Å from the Hg atom. This latter contact is less than the sum of the van der Waals radii of Hg and S (3.3 Å; Bondi, 1964). The Hg—S(1)—C(1)—S(2) torsion angle is −9 (1)° and the dihedral angle between the planes through the phenyl ring and the S_2CO moiety is 15.3°. Other geometric parameters for the molecule are as expected. The closest intermolecular contact involving the non-H atoms, 3.476 (4) Å, occurs between Hg and S(1′) (symmetry operation: $1-x, -y, -z$).

The Hg-atom coordination geometry found in the title complex closely resembles those found in the related species [MeHg(S_2COMe)] (Tiekink, 1986), [PhHg(S_2COMe)] and [PhHg(S_2CO^iPr)] (Tiekink, 1987). Indeed, similar structures have been found for the related dithiocarbamate species [MeHg(S_2CNEt_2)] (Chieh & Leung, 1976) and [PhHg(S_2CNEt_2)] (Tiekink, 1987), where the conformational flexibility of the xanthate ligand is not available.

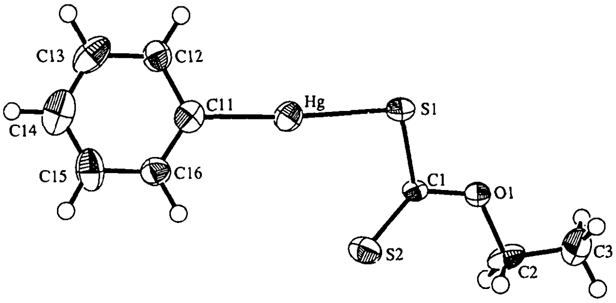


Fig. 1. Molecular structure of [PhHg(S_2COEt)] showing 50% probability displacement ellipsoids (Johnson, 1976).

Experimental

The title compound was prepared according to the method described in the literature (Tiekink, 1987). Crystals suitable for the X-ray analysis were grown by the vapour diffusion of diethyl ether into an acetonitrile solution of the compound (m.p. 394-395 K).

Crystal data

[Hg($C_3H_5OS_2$)(C_6H_5)]
M_r = 398.9
Orthorhombic
Pbca
a = 11.14 (1) Å
b = 27.092 (3) Å
c = 7.179 (4) Å
V = 2166 (2) Å^3
Z = 8
D_x = 2.446 Mg m^{-3}

Mo *K*α radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 37.2-43.8°
μ = 14.6 mm^{-1}
T = 293 K
Needle
0.11 × 0.16 × 0.48 mm
Pale yellow

Data collection

AFC-6*R* diffractometer
$\omega/2\theta$ scans [speed 32° $\min^{-1}$, width $(0.84 + 0.35\tan\theta)°$]
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.98$, $T_{\max} = 1.02$
2984 measured reflections
2984 independent reflections
1250 observed reflections [$I > 3\sigma(I)$]
$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 33$
$l = 0 \rightarrow 9$
3 standard reflections monitored every 400 reflections intensity variation: −3.57%

Refinement

Refinement on F
$R = 0.043$
$wR = 0.047$
$S = 1.86$
1250 reflections
119 parameters
H-atom positions calculated, U(H) based on $U_{\rm eq}$ of C atom to which H is bonded
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{\max} = 0.041$
$\Delta\rho_{\max} = 1.04$ e Å^{-3}
$\Delta\rho_{\min} = -1.42$ e Å^{-3}
Extinction correction: Zachariasen (1967), type II, Gaussian, isotropic
Extinction coefficient: 0.44425
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
Hg	0.48087 (5)	0.03983 (2)	0.25613 (9)	0.0370
S(1)	0.4764 (3)	−0.0462 (1)	0.2230 (6)	0.0379
S(2)	0.2686 (4)	−0.0188 (1)	0.4617 (5)	0.0397
O(1)	0.3234 (9)	−0.1053 (3)	0.325 (1)	0.0337
C(1)	0.345 (1)	−0.0575 (4)	0.342 (2)	0.0219
C(2)	0.228 (1)	−0.1270 (5)	0.430 (2)	0.0447
C(3)	0.233 (2)	−0.1805 (5)	0.397 (2)	0.0562
C(11)	0.493 (1)	0.1155 (5)	0.270 (2)	0.0343
C(12)	0.574 (2)	0.1396 (5)	0.150 (2)	0.0385
C(13)	0.581 (2)	0.1893 (6)	0.154 (2)	0.0512
C(14)	0.512 (2)	0.2153 (5)	0.278 (2)	0.0584
C(15)	0.436 (2)	0.1923 (5)	0.394 (2)	0.0523
C(16)	0.428 (2)	0.1424 (5)	0.391 (2)	0.0386

Table 2. *Selected geometric parameters* (Å, °)

Hg—S(1)	2.342 (3)	C(11)—C(12)	1.41 (2)
Hg—C(11)	2.06 (1)	C(11)—C(16)	1.34 (2)
S(1)—C(1)	1.73 (1)	C(12)—C(13)	1.35 (2)
S(2)—C(1)	1.60 (1)	C(13)—C(14)	1.37 (2)
O(1)—C(1)	1.32 (1)	C(14)—C(15)	1.34 (2)
O(1)—C(2)	1.43 (2)	C(15)—C(16)	1.35 (2)
C(2)—C(3)	1.47 (2)		
S(1)—Hg—C(11)	176.1 (4)	Hg—C(11)—C(16)	122 (1)
Hg—S(1)—C(1)	98.4 (4)	C(12)—C(11)—C(16)	119 (1)
C(1)—O(1)—C(2)	119 (1)	C(11)—C(12)—C(13)	119 (1)
S(1)—C(1)—S(2)	126.8 (8)	C(12)—C(13)—C(14)	119 (1)
S(1)—C(1)—O(1)	106.3 (9)	C(13)—C(14)—C(15)	121 (1)
S(2)—C(1)—O(1)	126 (1)	C(14)—C(15)—C(16)	119 (1)
O(1)—C(2)—C(3)	106 (1)	C(11)—C(16)—C(15)	121 (1)
Hg—C(11)—C(12)	118 (1)		

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

The Australian Research Council is thanked for the support of the crystallographic facility.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1099). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 862–864

Bis(4-aminopyridinium) Hexabromodicuprate(II) Diaquatetrabromodicopper(II)

HELEN PLACE AND ROGER WILLETT

Department of Chemistry, Washington State University, Pullman, Washington 99164-4630, USA

(*Received* 19 *May* 1992; *accepted* 18 *May* 1993)

Abstract

The structure of $(C_5H_7N_2)_2[Cu_2Br_6].[Cu_2Br_4(H_2O)_2]$ consists of stacks of alternating $Cu_2Br_6^{2-}$ anions and neutral $Cu_2Br_4(H_2O)_2$ molecules with the cations separating the stacks. The two chemically distinct species within the stack are both centrosymmetric quasi-planar bibridged dimers. The water molecules in the neutral dimers are located in *trans* positions.